

FEASIBILITY TESTS OF A RADIOISOTOPE-TYPE
ABLATION-MEASURING SYSTEM

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Presented at the Symposium on
Radioisotope Applications in Aerospace

GPO PRICE \$ _____

CFSTI PRICE(S) \$ _____

Hard copy (HC) 300

Microfiche (MF) 65

NASA TMX 57291

ff 653 July 65

Dayton, Ohio
February 15-17, 1966

FACILITY FORM 602	N 68-27574	
	(ACCESSION NUMBER)	(THRU)
	28	1
	(PAGES)	(CODE)
	TMX-57291	14
	(NASA CR OR TMX OR AD NUMBER)	(CATEGORY)

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ABSTRACT

The NASA-Langley Research Center has been studying the feasibility of making continuous measurements of the ablation of a spacecraft heat shield through the use of radioisotopes embedded in the heat shield. The technique consists of selecting two isotopes so that the depletion rate of one will correspond to the recession of the ablating surface and the depletion of the other is a measure of ablative material decomposition. A detector system capable of differentiating between energy levels monitors radiation from each of the two isotopes. Detected counts are integrated and supplied as outputs of the system in a form compatible with the spacecraft telemetry. The results of feasibility tests with Zr-Nb⁹⁵ and In^{114m} sources are presented and the problems encountered in incorporating radioactive compounds into ablation materials are discussed. The application of such a device in reentry vehicle systems may ultimately be limited by the characteristics of available isotopes and the suitability of existing detector systems.

INTRODUCTION

A manned spacecraft or unmanned space probe entering a planetary atmosphere has a high velocity and consequently a high kinetic energy which must be dissipated prior to impact. The vehicle is decelerated by collision with the atmosphere. In this process, it experiences

aerodynamic heating at rates such that surface temperatures are well above the melting or vaporization temperatures of available structural materials. To protect the spacecraft and its payload from this extreme heat, thermal protection systems have been developed.

Figure 1 is a photograph of a cross section of a typical charring ablation material representative of thermal protection systems currently being employed. As heat is applied, the ablation material protects the structure by decomposition or pyrolysis at a fairly low temperature giving off gases and leaving a carbon residue or "char." The gases percolate through the char, absorbing heat in the process. The gases are injected into the boundary layer, blocking convective heat transfer to the surface. Because of its very high melting temperature, the char reradiates a large amount of heat away from the ablator. The char finally recedes by sublimation or oxidation, or is mechanically removed by aerodynamic forces.

In order to effectively evaluate the performance of ablative thermal protection materials in ground tests or the flight environment, it is important to monitor the changes which occur in the material during the ablation process. These include the recession of the ablator surface and the recession of the interface between the virgin material and the decomposed material.

The design goals for an ablation sensing device are shown in figure 2. The first four of these goals are associated with any instrumentation used on a space vehicle. It must be lightweight, compact, rugged and have low power consumption. In addition, a continuous measurement of ablator surface position and char thickness at a single location on the

heat shield is desirable. In the past, it has been necessary to use two different instruments to make ablation measurements from which char thickness could be determined, and, because of space limitations, these devices often could not be located at the same point on the body. Further, it is desirable that in sensing ablation, a minimum of foreign material be imbedded in the heat shield to insure that the basic ablative properties of the heat shield are not changed. Finally, any sensing device should be unaffected by the external environment, particularly the ionized plasma surrounding the space vehicle during entry.

This paper will describe a technique being developed by the Langley Research Center, in-house and on contract to the Emerson Electric Company, which makes use of radioactive isotopes to continuously measure both surface recession and pyrolysis interface recession in a charring ablation material.

ABLATION SENSING TECHNIQUE

The sensing technique, illustrated in figure 3, makes use of the chemical properties of two gamma emitting isotopes. Compounds of these isotopes are chosen such that one decomposes or vaporizes at a temperature equivalent to the pyrolysis temperature of the ablator. The second is chosen to have a high decomposition temperature so that it will remain in the char until the char is oxidized or physically eroded. The compounds are uniformly impregnated into a 1/4-inch-diameter cylindrical plug of ablation material which is then installed in the space vehicle heat shield. During ablation, the depletion rate of one isotope (indicated by the ++ in fig. 3) will correspond to the rate of recession of the pyrolysis interface, while the depletion rate of the other isotope

(indicated by the in fig. 3) is a measure of ablator surface recession rate. A detector system capable of discriminating between energy levels monitors gamma radiation from each of the isotopes. Counting rates proportional to the ablation parameters are converted into a form suitable for the spacecraft telemetry system.

The measurement technique offers the distinct advantage of providing a measurement of both surface recession and pyrolysis interface recession at a single location on the spacecraft heat shield. This permits a straightforward determination of char thickness at that point. Also, the radioactive isotope technique will measure pyrolysis recession in ablation materials which are electrically conducting in their virgin state. In the past, the only technique available for measuring pyrolysis recession required that the virgin material have a high electrical resistivity while the char was highly conductive. In addition, this technique provides a continuous measurement and it requires only a minute amount of foreign material (isotope) imbedded in the ablation material.

ISOTOPE SELECTION

SELECTION CRITERIA

In implementing the proposed technique, criteria were first established for selecting suitable isotopes. These are summarized in figure 4. It was necessary that an isotope be selected which sublimates or decomposes at the pyrolysis temperature of the ablation materials of interest. The isotope for the surface recession measurement was required to have chemical properties such that it would remain in the heat shield

until removed by physical erosion or oxidation of the char layer. The two isotopes should emit gamma radiation of high enough energy levels to penetrate the ablation material and metallic structure with a minimum of absorption. Furthermore, the energy levels should be sufficiently different that they can readily be discriminated. Additional considerations would include the availability, cost, and specific activity of the isotope. Also, it is important that there be no chemical reaction between the radioactive compounds and the ablation material which would alter the thermal properties of the ablator. Finally, the half-life of the source must be considered as discussed later in this paper.

ABLATOR PYROLYSIS MEASUREMENT

The present study has centered around two ablation materials considered to be typical of materials currently being used. These are: phenolic nylon, a low-density composite of nylon powder and phenolic resin, and phenolic graphite, a high-density mixture of phenolic resin and graphite fibers. It was found experimentally through differential thermal analysis (DTA) and thermogravimetric analysis (TGA) that both of these ablators begin to decompose at a temperature of about 800° F.

Figure 5 lists radioactive compounds which appeared to have promise for the pyrolysis recession measurement. These included isotopes of mercury, iodine, iron, indium, scandium, and cobalt in compounds which decomposed at temperatures from 750° F to 1500° F. $\text{In}^{114\text{m}}$ immediately appeared attractive from both nuclear and chemical standpoints. It has a half-life of 50 days and 95% of all gamma radiation is emitted at an energy of 0.19 MeV with smaller energy peaks at 0.72 MeV, 0.57 MeV, and 1.3 MeV. In addition, it was found that many of the compounds of indium

volatize near 800° F. Also, a previous study had shown $\text{In}^{114\text{m}}\text{Cl}_3$ to be a reasonable indicator of pyrolysis recession in an ablation material similar in some respects to the two materials being studied in this program. For these reasons, $\text{In}^{114\text{m}}$ was chosen as the prime candidate isotope for the pyrolysis measurement.

SURFACE RECESSION MEASUREMENT

Figure 6 lists compounds which, from a survey of available isotopes, appeared to have promise for the surface recession measurement. These included compounds of zirconium, niobium, tantalum, cerium, and europium. All are refractory compounds which because of their high decomposition temperature, should remain in the heat shield until removed by erosion or oxidation of the char layer.

The Zr-Nb⁹⁵ isotopes appeared most attractive from a nuclear point of view, since the gamma activity is almost entirely (99%) contained between 0.72 and 0.77 MeV. This would permit relatively simple discrimination from the 0.19 MeV $\text{In}^{114\text{m}}$ radiation. The zirconium and niobium activities appear together since Nb⁹⁵ is a daughter product of Zr⁹⁵. At equilibrium, the Zr-Nb decays with a half-life of 65 days.

IMPREGNATION TECHNIQUES

Concurrent with the survey of isotopes, an investigation was made of techniques for inserting isotopes into ablation materials. With a guideline of a minimum of foreign material in the heat shield, methods of impregnating radioactive compounds directly into the ablation material were studied. The phenolic nylon ablator with a density of 0.6g/cc

is typical of many low-density ablation materials. It was found that these materials could be impregnated by immersing the ablator plug directly into a solution containing the radioactive compounds. However, with the phenolic graphite ablator, because of its higher density (1.3g/cc) and hardness, it was found necessary to soak the uncured ablation material in the radioactive compound in solution. The ablator was then molded into disks which were machined into cylindrical plugs. The distribution of activity in sensor plugs produced using these techniques was measured experimentally and found to be reasonably uniform. Any nonuniformity was noted and taken into account later in analysis of test data.

EXPERIMENTAL RESULTS

The technique used to evaluate isotopes as indicators of heat shield ablation was to test them in a heating environment using an oxygen-acetylene torch as the heat source. Usually, ablation material tests are conducted in a plasma arc facility where one or more of the important characteristics of entry heating can be simulated. However, the lack of an arc facility suitable for handling radioactive isotopes necessitated the use of the oxygen-acetylene torch to provide a source of heat.

Calibration curves of count rate versus material thickness were first established for each isotope and ablator. One-fourth-inch-diameter plugs of ablator impregnated with approximately 20 microcuries of activity were sliced into ten 1/10-inch-thick disks. The 10 disks were stacked in a test specimen approximately 1 inch from a scintillation detector

containing a 3/4-inch- by 1/2-inch-diameter NaI crystal. The change in count rate was measured as the disks were removed one at a time. These data established count rate versus ablator thickness calibration curves for the system. The calibration curve for $\text{In}^{114\text{m}}$ in the phenolic nylon ablator is shown in figure 7.

The setup for conducting ablation tests is shown in figure 8. The test specimen is fabricated by bonding a 1/4-inch-diameter plug of ablator impregnated with approximately 20 microcuries of each isotope into a 1-inch-diameter cylinder of the same material. The specimen is mounted in the same configuration as for calibration to maintain the source-detector geometry. The desired heating condition is established by adjusting the oxygen-acetylene pressure ratio and the distance between heat source and test specimen. Before and after the test, the count rate and background are read in each discrimination "window" with a single channel analyzer. The specimen is subjected to the heating environment for a specified length of time. During the test, the change in activity as the specimen ablates is monitored with a rate-meter. The resulting test data are corrected by subtracting the measured background level. The data from the $\text{In}^{114\text{m}}$ source are further corrected by subtracting the count rate due to Compton scatter in the lower window from the higher energy radiation. Using these corrected data and the calibration curve, the final ablator thickness can be determined. This "predicted" thickness is then compared with physical measurements of the actual specimen thickness to determine the measurement error.

Figure 9 illustrates the results of a test of $\text{In}^{114\text{m}}$ measuring the pyrolysis recession of the phenolic nylon ablator. Ablator thickness, obtained from the count rate and calibration data, is plotted versus test time in seconds. The predicted final thickness from the data was 0.22 inch. Physical measurements following the test showed the actual thickness of virgin material to be 0.28 inch. This results in a measurement error of 0.06 inch.

An illustration of the results of a test of Zr-Nb^{95} measuring the recession of the phenolic nylon ablator surface is shown in figure 10. Once again, ablator thickness, obtained from the count rate and calibration data, is plotted versus test time. For this test, the predicted thickness of 0.22 inch when compared with the actual measured specimen thickness of 0.18 inch resulted in a measurement error of 0.04 inch.

The results of a series of ablation tests indicate that the dual ablation-measuring technique utilizing $\text{In}^{114\text{m}}$ and Zr-Nb^{95} sources is capable of measuring both surface recession and pyrolysis recession of an ablating heat shield. However, it would be desirable if isotopes could be found which do not possess some of the characteristics of the existing sources, such as the indium energy peak at 0.72 MeV within the zirconium-niobium window. Furthermore, before an instrumentation system of this nature can be utilized effectively in a flight vehicle, several requirements must be met.

FLIGHT-SYSTEM REQUIREMENTS

The requirements for a flight instrumentation system are outlined in figure 11. The choice of isotope for a flight system is limited by the

geometry of the payload to the extent that the energy levels must be high enough that the radiation pass through the ablation material and metallic vehicle structure with a minimum of absorption. The complex operations surrounding the launch of a space vehicle require that the source may have to be installed in the payload several days or even weeks prior to launch. This fact plus the possibility of extended delays during launch operations preclude the use of short half-lived isotopes in the system as presently configured. A reasonable minimum source half-life of 50 days has been chosen. In addition, should the launch be postponed or canceled, the source must be capable of being removed. A final guideline in selecting a source for a flight application is that a minimum of activity be used in the heat shield to reduce the exposure to gamma radiation of personnel working in the vicinity of the payload.

It is important that the detection system detect radiation efficiently to be consistent with the requirement for a minimum of activity in the heat shield. Since the detector assembly would be mounted near the ablating heat shield in an area of the payload where space is generally at a premium, it should occupy a minimum volume. However, one of the most stringent requirements imposed upon the detector is that it be rugged. A payload launched on a small solid-propellant booster in an earth reentry flight test may experience accelerations up to 150g and shocks up to 250g for 15 milliseconds. A scintillation crystal-photomultiplier detector assembly rugged enough to survive this environment might occupy a volume of as much as 25 cubic inches. Hopefully, new detectors will be developed which are small, rugged, and efficient detectors of gamma radiation.

A final requirement which must be met in developing this system for flight is to conduct the necessary flight qualification tests including a number of tests in a simulated entry heating environment. Since a plasma arc facility with the capability of handling millicurie levels of activity is not available, it has been necessary to utilize the oxygen-acetylene torch extensively as a testing tool and screening device. However, before this system can be used to effectively measure the in-flight characteristics of ablation materials, its performance must first be evaluated in an environment which is more representative of entry heating.

CONCLUSIONS

Tests have been conducted to determine the feasibility of utilizing gamma radiation from isotopes to measure the thermal and physical changes that occur in an ablating thermal protection material. The results indicate that simultaneous measurements of surface recession and pyrolysis recession can be made at a single location with only a minute amount of foreign material imbedded in the ablator. However, the ability of this technique to measure the in-flight characteristics of an ablating heat shield may ultimately be limited by the characteristics of available isotopes. Also it is hoped that new detectors will be developed that can efficiently detect gamma radiation, are small in size, and rugged enough to withstand the severe environment associated with space vehicles entering a planetary atmosphere.

ACKNOWLEDGMENT

The author acknowledges the contributions of the Emerson Electric Company - Electronics and Space Division to all phases of this work under NASA contracts NAS1-5342 and NAS9-2610.

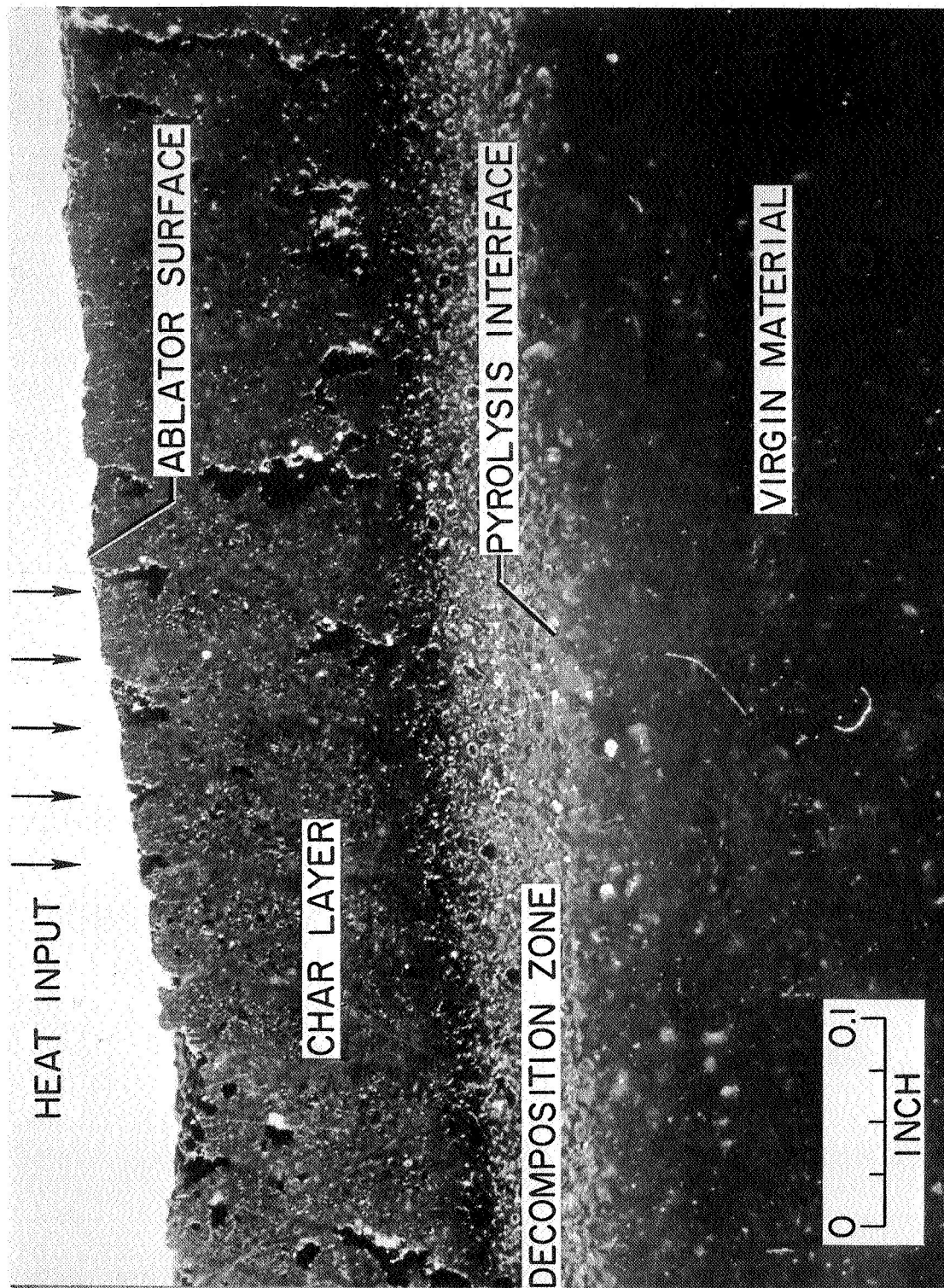


Figure 1.- Cross section, typical charring ablation material.

1. SMALL VOLUME
2. RUGGED
3. LIGHT WEIGHT
4. LOW POWER CONSUMPTION
5. CONTINUOUS SURFACE POSITION AND CHAR THICKNESS MEASUREMENT
6. MINIMUM OF FOREIGN MATERIAL IN HEAT SHIELD
7. UNAFFECTED BY PLASMA SURROUNDING VEHICLE

Figure 2.- Design goals for an ablation sensor.

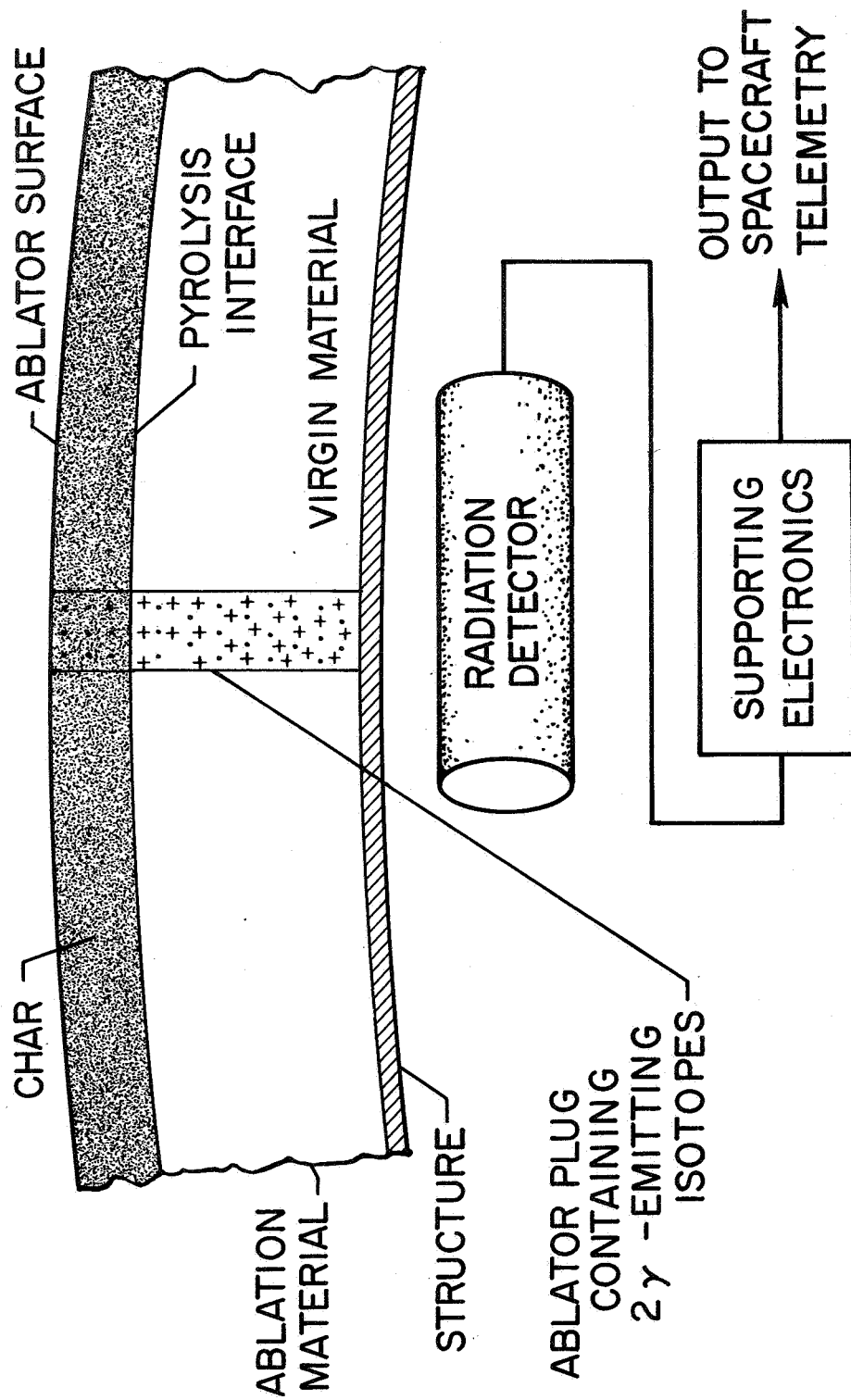


Figure 3.- Ablation sensing technique.

1. ONE RADIOACTIVE COMPOUND SUBLIMES AT DECOMPOSITION TEMPERATURE OF ABLATOR
2. SECOND COMPOUND REMAINS IN CHAR
3. GAMMA EMITTERS
4. ENERGY LEVELS READILY DISCRIMINATED
5. AVAILABILITY
6. COST
7. SPECIFIC ACTIVITY
8. NO CHEMICAL REACTIONS WITH ABLATOR
9. HALF LIFE

Figure 4.- Criteria for selecting isotopes.

COMPOUND	DECOMPOSITION TEMP	ISOTOPE	HALF LIFE	PRINCIPLE γ RADIATION ENERGY
Hg ₂ Cl ₂	750°F	Hg ^{197m}	24 HOURS	0.16 Me V
Hg S	1080	Hg ¹⁹⁷	65 HOURS	0.08
NaI	1140	I ¹³¹	8 DAYS	0.08-0.36
FeCl ₂	1100	Fe ⁵⁹	45 DAYS	0.19, 1.3
Hg S	1080	Hg ²⁰³	47 DAYS	0.28
InCl ₃	820	In ^{114m}	50 DAYS	0.19
NH ₄ I	750-1000	I ¹²⁵	60 DAYS	0.04
ScCl ₃	1000	Sc ⁴⁵	84 DAYS	1.1
CoCl ₃	1500	Co ⁶⁰	5.3 YEARS	1.2-1.3

Figure 5.- Radioactive compounds considered for pyrolysis measurement.

<u>COMPOUND</u>	<u>DECOMPOSITION TEMP.</u>	<u>ISOTOPE</u>	<u>HALF LIFE</u>	<u>PRINCIPLE γ RADIATION ENERGY</u>
Zr - Nb C	6400-7000° F	Zr - Nb ⁹⁵	65 DAYS	0.72 - 0.77 MeV
Ta C	7400	Ta ¹⁸²	115 DAYS	0.1-1.2
Ce C	6130	Ce ¹⁴⁴	285 DAYS	0.13
Eu C	5800	Eu ¹⁵²	13 YEARS	0.1-1.2

Figure 6.- Radioactive compounds considered for surface recession measurement.

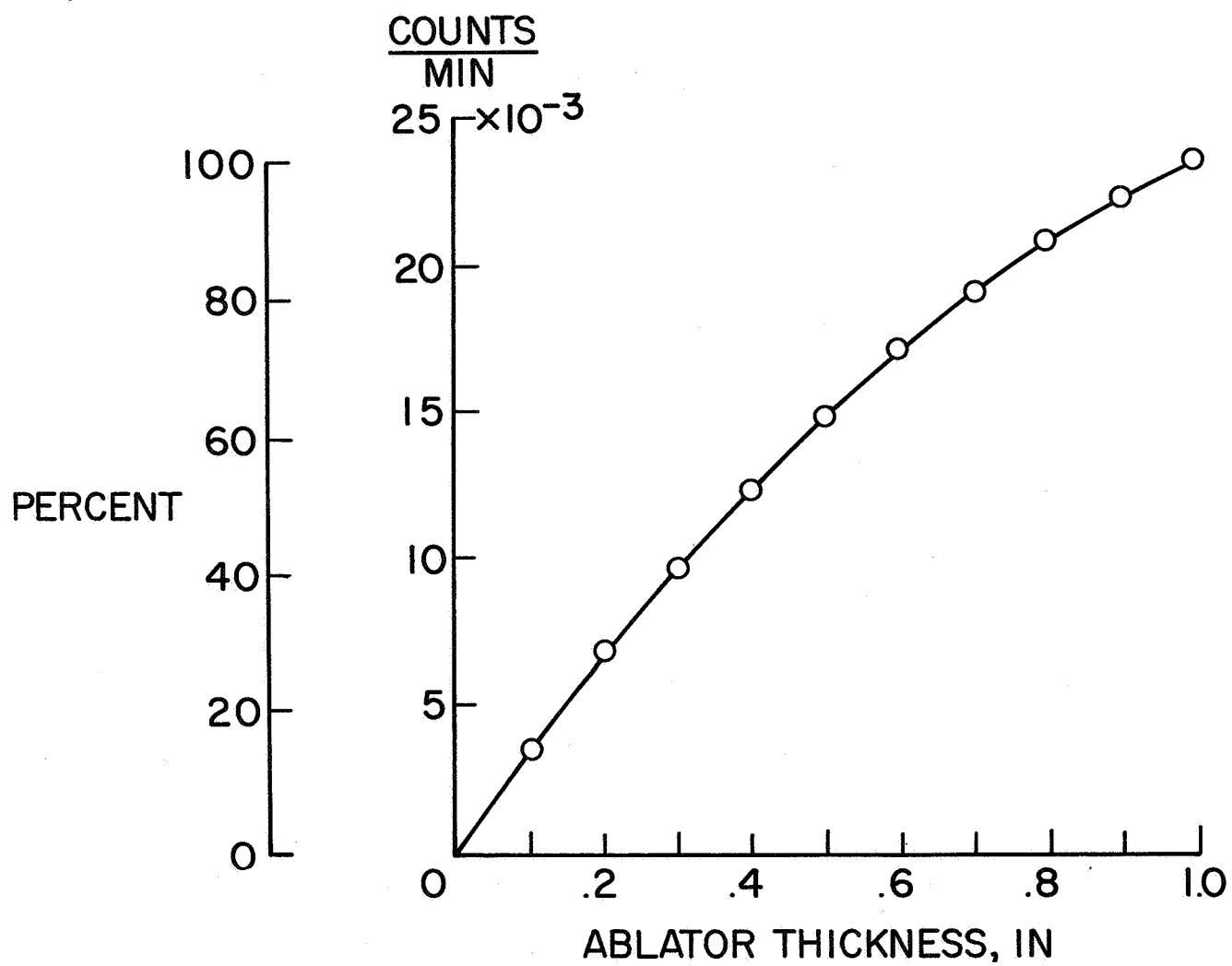


Figure 7.- Calibration curve - $\text{In}^{114\text{m}}$, phenolic nylon ablator.

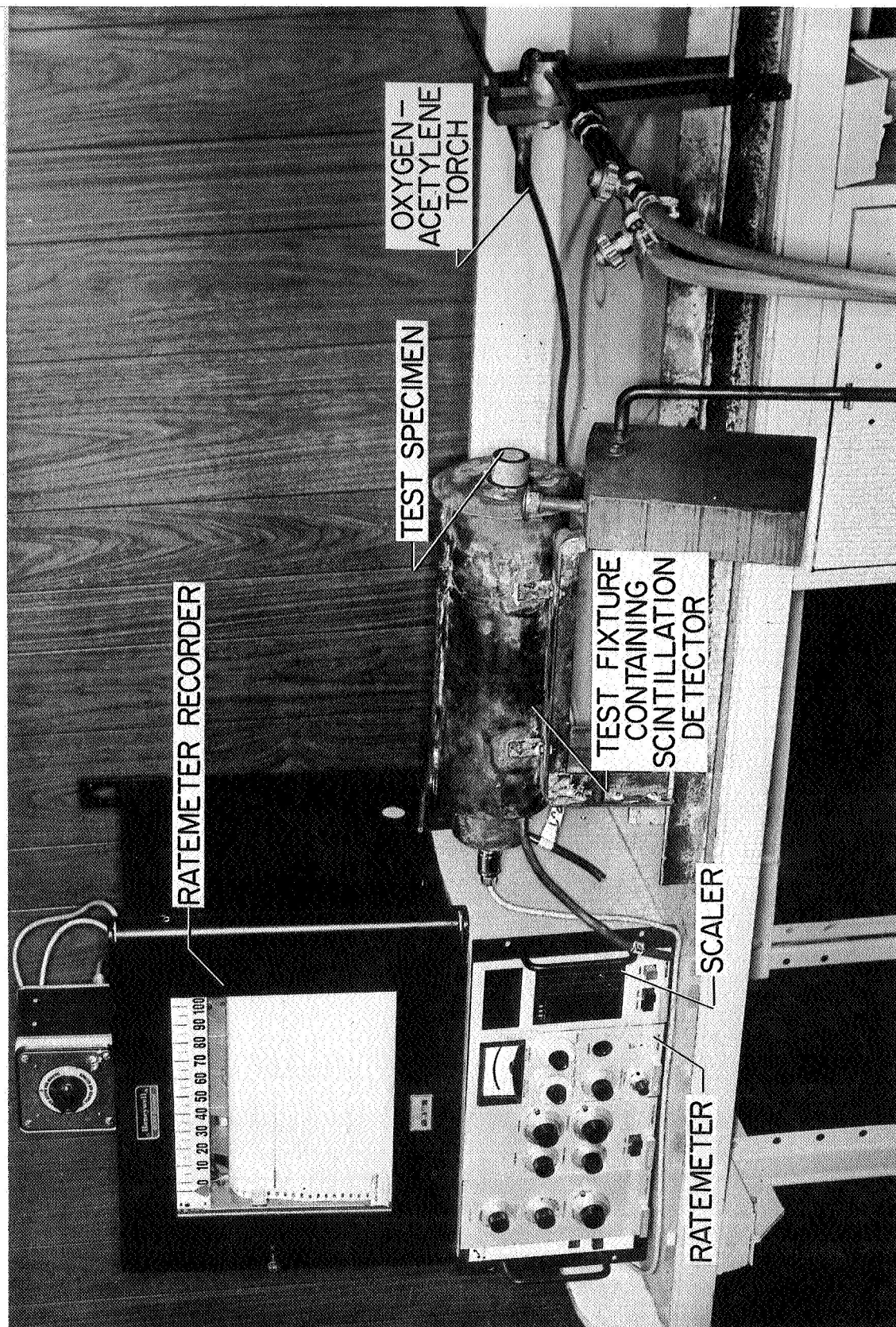


Figure 8.- Ablation test setup.

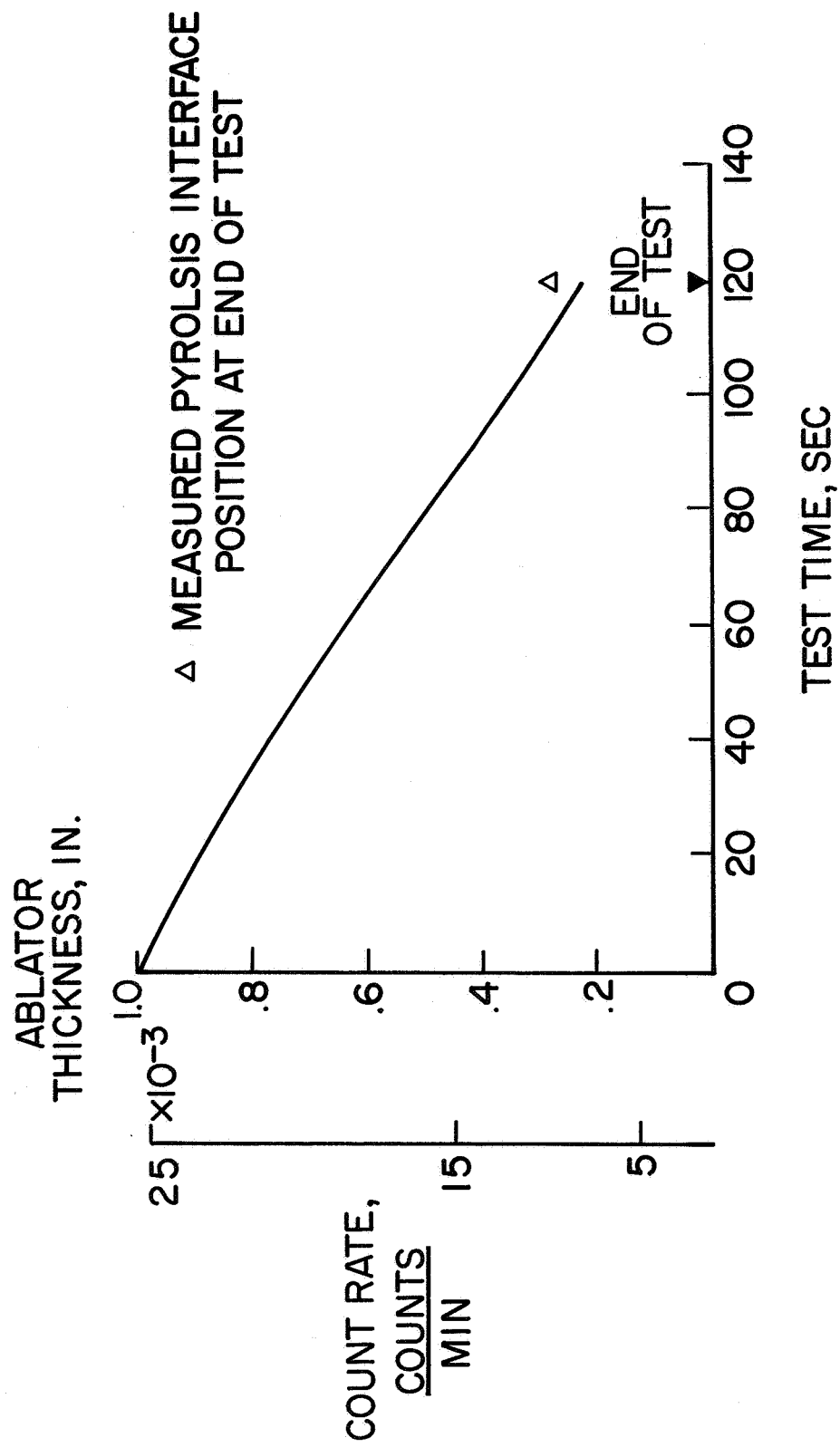


Figure 9.- Pyrolysis interface recession test #PN-3, phenolic nylon ablator.

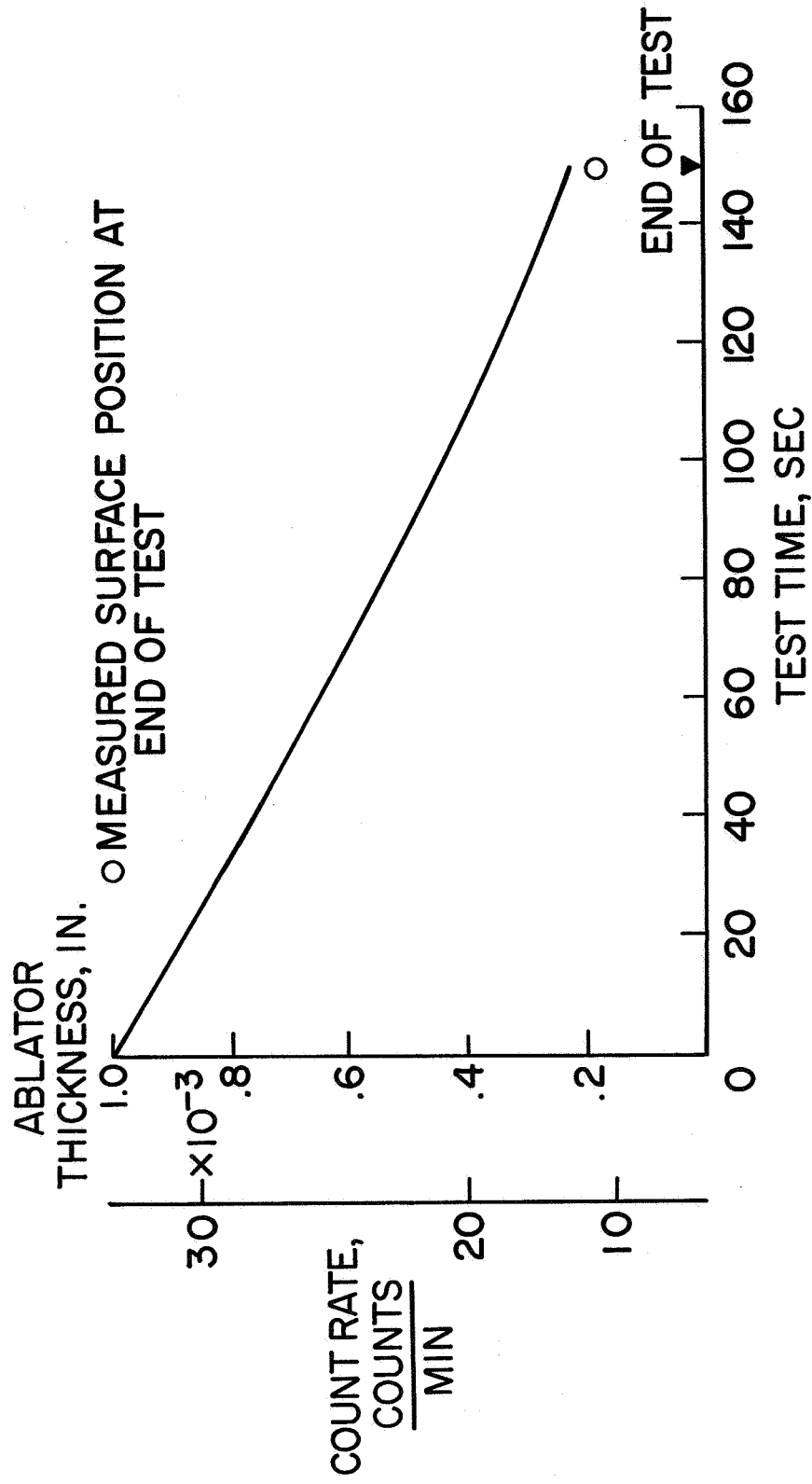


Figure 10.- Surface recession test #PN-4, phenolic nylon ablator.

- SOURCE - RADIATION MUST PENETRATE HEAT SHIELD
AND SUBSTRUCTURE

MINIMUM HALF LIFE OF 50 DAYS

REMOVEABLE

MINIMUM ACTIVITY IN HEAT SHIELD

- DETECTION SYSTEM

EFFICIENT

MINIMUM VOLUME

RUGGED

- PRE-FLIGHT TEST PROGRAM

Figure 11.- Flight system requirements.